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Silica Supported Perchloric acid ($HClO_4$-SiO_2) Catalyzed Synthesis of 8,10-Dimethyl-8,12-dihydro-12-aryl-9*H*-naphtho-[1',2':5,6]pyrano[2,3-d]pyrimidine-9,11(10*H*)-diones Under Solvent-Free Conditions

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The reaction of β-naphthol with arylaldehydes and 1,3-dimethylbarbituric acid in the presence of $HClO_4$-SiO_2 (3 mol %) under solvent-free conditions led to 8,10-dimethyl-8,12-dihydro-12-aryl-9*H*-naphtho-[1',2':5,6]pyrano[2,3-d]pyrimidine-9,11(10*H*)-diones in good yield.

Key Words: Naphtho[1',2':5,6]pyrano[2,3-d]pyrimidine, β-Naphthol, $HClO_4$-SiO_2, 1,3-Dimethylbarbituric acid.

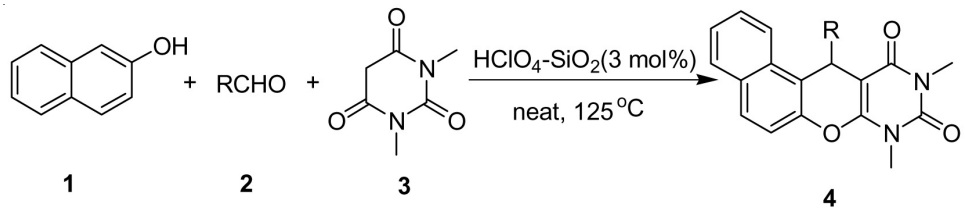
INTRODUCTION

Multicomponent reactions have attracted considerable attention since they are performed without need to isolate any intermediate during their processes which reduces time and saves both energy and raw materials[1]. They have merits over two-component reactions in several aspects including the simplicity of a one-pot procedure, possible structural variations and building up complex molecules.

Natural compounds possessing naphthopyran moiety have been attracted by their antimicrobial[2], antitumor[3], antifungal[4], cytotoxic[5], antioxidative and 5-lipoxygenase inhibitory activity[6]. A variety of naphthopyran derivatives have been isolated and identified as natural phytochemicals[7]. A plethora of biological activities have also been associated with a large number of synthetic naphthofuran analogs[8]. Pyrano[2,3-*d*]pyrimidines and chromeno[2,3-*d*]pyrimidines are two 'privileged medicinal scaffolds' which are used for the development of pharmaceutical agents of various applications. Compounds with these motif show a wide range of pharmacological activities such as antiviral[9], antimicrobial[10], antifungal[11], anticonvulsant and analgesic activities[12]. Moreover, they are also useful reagents in organic synthesis, for example, 5-deaza-10-oxaflavin possess a strong function to oxidize alcohols to the corresponding carbonyl compounds[13].

In recent years, the use of heterogeneous catalysts has received considerable interest in various disciplines including organic synthesis. They are advantageous over their homogeneous counterparts due to the prime advantage that in most of the cases the catalyst can be recovered easily and reused. Silica supported perchloric acid ($HClO_4$-SiO_2) has been used as an efficient heterogeneous catalyst for many

organic transformations because of its low cost, ease of preparation, catalyst recycling and ease of handling[14]. We now report a simple and efficient route to synthesis of 8,10-dimethyl-8,12-dihydro-12-aryl-9*H*-naphtho[1',2':5,6]pyrano[2,3-d]pyrimidine-9,11(10*H*)-diones using $HClO_4$-SiO_2 as an efficient and environmentally benign catalyst under solvent-free conditions (**Scheme-I**).



Scheme-I

EXPERIMENTAL

NMR spectra were determined on Bruker AV-400 spectrometer in $CDCl_3$ and were expressed in δ values relative to tetramethyl silane, coupling constants (*J*) were measured in Hz; elemental analysis were recorded on a Vario ELIII elemental analyzer; melting points were determined on a Mel-Temp capillary tube apparatus and were uncorrected; commercially available reagents were used throughout without further purification unless otherwise stated.

General procedure for the preparation of compound 4: To a mixture of 2-naphthol (1 mmol), aldehydes (1 mmol) and 1,3-dimethylbarbituric acid (1 mmol), $HClO_4$-SiO_2 (60 mg, 0.03 mmol) was added. The mixture was stirred at 125 ºC for an appropriate time (Table-1). After completion of the reaction (TLC), 10 mL CH_2Cl_2 was added to the reaction mixture and the catalyst was recovered by filteration. The organic layer was dried over $MgSO_4$, the solvent was evaporated. Products **4** were purified by silica gel column chromatography using CH_2Cl_2 as eluent.

8,10-Dimethyl-8,12-dihydro-12-phenyl-9*H*-naphtho[1',2':5,6]pyrano[2,3-d]pyrimid ine-9,11(10*H*)-diones (4a): ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 7.96 (d, *J* = 8.4 Hz, 1H), 7.85 (t, *J* = 8.6 Hz, 2H), 7.50-7.38 (m, 5H), 7.23 (t, *J* = 7.6 Hz, 2H), 7.13 (t, *J* = 14.4 Hz, 1H), 5.83 (s, 1H,), 3.62 (s, 3H), 3.35 (s, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 161.9, 152.2, 150.7, 147.2, 143.9, 131.8, 130.9, 129.5, 128.5, 128.4, 128.2, 127.4, 126.8, 125.5, 123.9, 91.5, 36.0, 29.0, 28.2. Anal. calcd. for $C_{23}H_{18}N_2O_3$: C 74.58; H 4.90; N 7.56, found: C 74.50; H4.88; N 7.68.

8,10-Dimethyl-8,12-dihydro-12-(4-chlorophenyl)-9*H*-naphtho[1',2':5,6]-pyrano[2,3-d]pyrimidine-9,11(10*H*)-diones (4b): ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 7.89-7.83 (m, 3H), 7.50-7.40 (m, 3H), 7.31 (d, *J* = 8.4 Hz, 2H), 6.90 (d, *J* = 8.4 Hz, 2H), 5.78 (s, 1H,), 3.69 (s, 3H), 3.35 (s, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 161.9, 152.2, 150.6, 147.1, 142.3, 132.5, 131.8, 130.7, 129.8, 129.6, 128.6, 127.6, 125.6, 123.7, 116.8, 116.3, 91.0, 35.5, 29.1, 28.2. Anal. calcd. for $C_{23}H_{17}NO_3Cl$: C 68.23; H 4.23; N 6.92, found: C 68.36; H 4.15; N 6.99.

TABLE-1
TEMPERATURE OPTIMIZATION FOR THE SYNTHESIS OF 8,10-DIMETHYL-8,12-DIHYDRO-12-ARYL-9*H*-NAPHTHO [1',2':5,6]PYRANO [2,3-*d*]PYRIMI-DINE-9,11(10*H*)-DIONES*

Entry	Temperature (ºC)	Yield (%)**
1	Room temperature	0
2	85	54
3	90	59
4	95	61
5	100	66
6	105	71
7	110	74
8	115	76
9	120	82
10	125	88
11	130	86
12	135	88
13	140	85

*Reaction conditions: 2-naphthol (1 mmol); benzaldehyde (1 mmol); 1,3-dimethylbarbituric acid (1 mmol); $HClO_4$-SiO_2 (0.03 mmol); solvent-free; 1 h. **Isolated yield after chromatographic purification.

8,10-Dimethyl-8,12-dihydro-12-(4-methoxyphenyl)-9*H*-naphtho-[1',2':5,6]-pyrano[2,3-d]pyrimidine-9,11(10H)-diones (4c): ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 7.96 (d, *J* = 8.4 Hz, 1H), 7.83 (t, *J* = 7.2 Hz, 2H), 7.50-7.39 (m, 3H), 7.31-7.28 (m, 2H), 6.74 (d, *J* = 8.4 Hz, 2H), 5.78 (s, 1H,), 3.69 (s, 3H), 3.62 (s, 3H), 3.35 (s, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 164.7, 162.0, 158.2, 152.1, 150.6, 147.1, 136.2, 131.8, 130.9, 129.4, 129.2, 128.5, 127.4, 125.5, 124.0, 117.6, 116.3, 113.9, 113.8, 91.6, 55.1, 35.1, 29.0, 28.2. Anal. calcd. for $C_{24}H_{20}N_2O_4$: C 71.99; H 5.03; N 7.00 found: C 72.14; H 5.00; N 7.12.

8,10-Dimethyl-8,12-dihydro-12-(4-tolyl)-9*H*-naphtho[1',2':5,6]pyrano[2,3-d]pyrimidine-9,11(10*H*)-diones (4d): ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 7.97 (d, *J* = 8.0 Hz, 1H), 7.84 (t, *J* = 7.8 Hz, 2H), 7.50-7.40 (m, 4H), 7.31-7.27 (m, 2H), 7.02 (d, *J* = 8.0 Hz, 1H), 5.79 (s, 1H), 3.62 (s, 3H), 3.35 (s, 3H), 2.23 (s, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 161.9, 152.1, 150.7, 147.1, 141.0, 136.3, 134.3, 131.8, 130.9, 129.4, 129.1, 128.5, 128.1, 127.4, 125.5, 123.9, 117.5, 116.3, 91.6, 35.6, 29.0, 28.2, 21.0. Anal. calcd. for $C_{24}H_{20}N_2O_3$: C 74.98; H 5.24; N 7.29. found: C75.08; H 5.20; N 7.33.

8,10-Dimethyl-8,12-dihydro-12-(4-nitrophenyl)-9*H*-naphtho[1',2':5,6]-pyrano[2,3-d]pyrimidine-9,11(10*H*)-diones (4e): ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 8.28 (d, *J* = 8.4 Hz, 2H), 8.07 (d, *J* = 8.8 Hz, 1H), 7.97-7.81 (m, 5H), 7.55 (d, *J* = 8.8 Hz, 2H), 5.92 (s, 1H,), 3.64 (s, 3H), 3..35 (s, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 161.6, 159.7, 155.2, 150.9, 147.2, 139.0, 132.2, 130.3, 129.2, 128.8, 127.8, 123.7, 123.4, 123.1, 116.4, 115.81, 90.0, 36.0, 29.2, 28.2. Anal. calcd. for $C_{23}H_{17}N_3O_5$: C 66.50; H 4.12; N 10.12 found: C 66.55; H 4.08; N 10.18.

8,10-Dimethyl-8,12-dihydro-12-(4-fluorophenyl)-9*H*-naphtho-[1',2':5,6]-pyrano[2,3-d]pyrimidine-9,11(10*H*)-diones (4f): ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 7.92-7.84 (m, 3H), 7.51-7.33 (m, 5H), 6.90 (t, *J* = 8.6 Hz, 2H), 5.81 (s, 1H,), 3.63 (s, 3H), 3.36 (s, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 162.0, 160.3, 152.2, 150.6, 147.1, 139.6, 131.8, 130.8, 129.8, 129.7, 128.6, 127.5, 125.6, 123.8, 117.1, 116.3, 115.4, 115.1, 91.2, 35.3, 29.0, 28.2. Anal. calcd. for $C_{23}H_{17}NO_3F$: C 71.13; H 4.41; N 7.21. found: C 71.20; H 4.35; N 7.29.

8,10-Dimethyl-8,12-dihydro-12-(3-nitrophenyl)-9*H*-naphtho-[1',2':5,6]-pyrano[2,3-d]pyrimidine-9,11(10*H*)-diones (4g): ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 8.17 (d, *J* =7.6 Hz, 1H), 8.06 (s, 1H), 8.01-7.42 (m, 8H), 5.93 (s, 1H,), 3.69 (s, 3H), 3..35 (s, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 161.5, 159.9, 155.3, 150.9, 147.8, 145.8, 137.7, 134.8, 134.2, 130.3, 129.0, 126.7, 126.2, 123.4, 122.1, 122.0, 121.1, 116.5, 115, 90.0, 36.0, 29.4, 28.6. Anal. calcd. for $C_{23}H_{17}N_3O_5$: C 66.50; H 4.12; N 10.12 found: C 66.42; H 4.24; N 10.20.

8,10-Dimethyl-8,12-dihydro-12-(2-chlorophenyl)-9*H*-naphtho-[1',2':5,6]-pyrano[2,3-d]pyrimidine-9,11(10*H*)-diones (4h): ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 8.20, (d, *J* = 8.4 Hz, 1H), 7.82 (t, *J* = 8.2 Hz, 2H), 7.53 (t, *J* = 7.6 Hz, 1H), 7.51-7.29 (m, 6H). 6.13 (s, 1H,), 3.66 (s, 3H), 3.34 (s, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 161.7, 152.5, 150.6, 147.0, 141.2, 133.1, 131.6, 131.5, 131.2, 130.1, 129.8, 128.5, 128.1, 127.5, 127.1, 125.5, 124.1, 116.9, 116.3, 90.5, 34.2, 29.1, 28.2. Anal. calcd. for $C_{23}H_{17}NO_3Cl$: C 68.23; H 4.23; N 6.92. found: C 68.41; H 4.20; N 7.01.

8,10-Dimethyl-8,12-dihydro-12-(2,4-dichlorophenyl)-9*H*-naphtho-[1',2':5,6]pyrano[2,3-d]pyrimidine-9,11(10*H*)-diones (4i): ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 8.10 (d, *J* = 8.8 Hz, 1H), 7.84 (t, *J* = 7.8 Hz, 2H), 7.56-7.52 (m, 1H), 7.48-7.44 (m, 1H), 7.36 (d, *J* = 8.8 Hz, 1H), 7.33 (d, *J* = 2 Hz, 1H), 7.22 (d, *J* = 8 Hz, 1H), 7.08 (dd, *J* = 8.4 Hz, 1H), 6.08 (s, 1H,), 3.65 (s, 3H), 3.34 (s, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 161.7, 152.5, 150.6, 147.0, 139.8, 133.8, 133.1, 132.4, 131.7, 131.0, 129.8, 127.7, 127.5, 125.6, 123.8, 90.0, 33.9, 29.1, 28.2. Anal. calcd. for $C_{23}H_{16}N_2O_3Cl_2$: C 62.88; H 3.67; N 6.38. found: C 62.91; H 3.60; N 6.45.

8,10-Dimethyl-8,12-dihydro-12-(3,4-dichlorophenyl)-9*H*-naphtho-[1',2':5,6]pyrano[2,3-d]pyrimidine-9,11(10*H*)-diones (4j): ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 7.91-7.85 (m, 3H), 7.54-7.28 (m, 6H), 5.79 (s, 1H), 3.64 (s, 3H), 3.36 (s, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 161.8, 152.3, 150.5, 147.1, 144.0, 132.5, 131.8, 130.9, 130.6, 130.2, 130.1, 128.7, 127.9, 127.7, 125.7, 123.6, 116.3, 116.1, 90.4, 35.4, 29.1, 28.2. Anal. calcd. for $C_{23}H_{16}N_2O_3Cl_2$: C 62.88; H 3.67; N 6.38. found: C 62.80; H 3.65; N 6.32.

RESULTS AND DISCUSSION

Initially, to optimize the reaction temperature, the reaction of 2-naphthol (1 mmol) with benzaldehyde (1 mmol) and 1,3-dimethylbarbituric acid was studied under solvent-free conditions in the presence of 3 mol % $HClO_4$-SiO_2 at different temperatures. The results are summarized in Table-1. As shown in Table-1, the reaction at 125 °C proceeded in highest yield.

To optimize the catalyst loading, 0, 1, 2, 3, 4 and 5 mol % of was tested, respectively. The results are summarized in Table-2. A 3 mol % loading of $HClO_4$-SiO_2 was sufficient to push the reaction forward and 2 mol % of $HClO_4$-SiO_2 was not enough. Higher amounts of $HClO_4$-SiO_2 did not lead to significant change in the reaction yields.

TABLE-2
AMOUNTS OF CATALYST OPTIMIZATION FOR THE SYNTHESIS OF 8,10-DIMETHYL-8,12-DIHYDRO-12-ARYL-9*H*-NAPHTHO[1',2':5,6]PYRANO [2,3-*d*]PYRIMIDINE-9,11(10*H*)-DIONES*

Entry	$HClO_4$-SiO_2 (mol) (%)	Yield (%)**
1	0	0
2	1	58
3	2	79
4	3	88
5	4	87
6	5	85

*Reaction conditions: 2-naphthol (1 mmol); benzaldehyde (1 mmol); 1,3-dimethylbarbituric acid (1 mmol); solvent-free;125 ºC; 2 h. **Isolated yield after chromatographic purification.

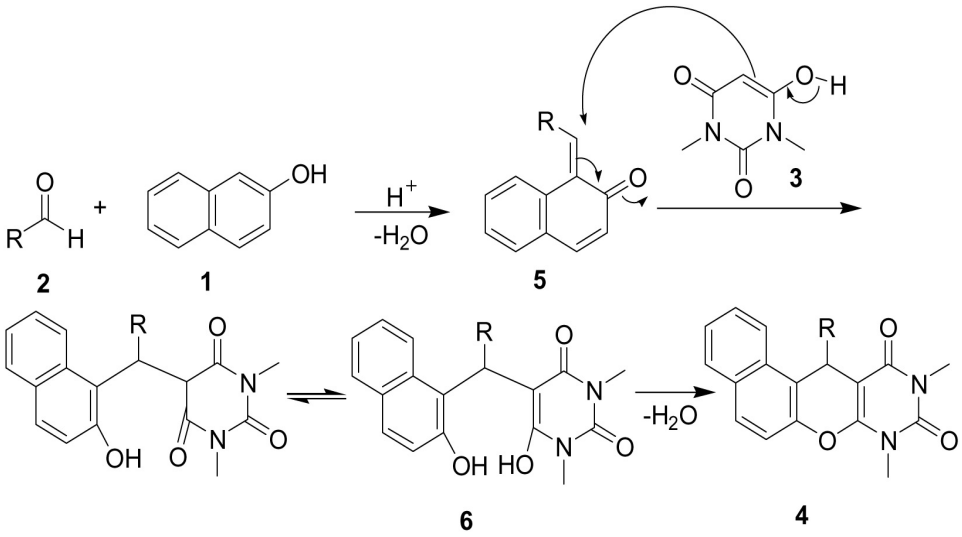
Based on the optimized reaction conditions, a range of 8,10-dimethyl-8,12-dihydro-12-aryl-9*H*-naphtho[1',2':5,6]pyrano[2,3-d]pyrimidine-9,11(10*H*)-diones (**4**) was synthesized by the reaction of 2-naphthol (**1**, 1 mmol) with arylaldehydes (**2**, 1 mmol) and 1,3-dimethylbarbituric acid (**3**, 1 mmol). The reaction proceeded at 125 ºC within 2 h in excellent yields after the addition of the acid catalyst $HClO_4$-SiO_2 (see Table-3). In these experiments the catalyst was isolated by filtration and could be reloaded with fresh reagents for further runs, thus, recyclization of catalyst is possible without significant loss of activity (Table-3, entry 1).

TABLE-3
SYNTHESIS OF 8,10-DIMETHYL-8,12-DIHYDRO-12-ARYL-9*H*-NAPHTHO[1',2':5,6]PYRANO [2,3-*d*]PYRIMIDINE-9,11(10*H*)-DIONES*

Entry	Aldehyde	Time (h)	Product	Yield (%)	m.p. (ºC)
1	C_6H_5	2.0	**4a**	88(86,83,83)**	220-222
2	4-Cl-C_6H_4	1.0	**4b**	91	272-273
3	4-Br-C_6H_4	2.0	**4c**	85	245-246
4	4-Me-C_6H_4	2.0	**4d**	84	188-190
5	4-NO_2-C_6H_4	1.5	**4e**	94	280-282
6	4-F-C_6H_4	1.5	**4f**	90	296-298
7	2-NO_2-C_6H_4	2.0	**4g**	89	278-280
8	2-Cl-C_6H_4	1.0	**4h**	95	266-268
9	2,4-Cl_2-C_6H_3	1.0	**4i**	93	220-221
10	3,4-Cl_2-C_6H_3	1.0	**4j**	92	246-248

*Reaction conditions: 2-naphthol (1 mmol); arylaldehyde (1 mmol); 1,3-dimethylbarbituric acid (1 mmol); $HClO_4$-SiO_2 (0.03 mmol); solvent-free; 125 ºC. **Isolated yield after recycling of catalyst.

A tentative mechanism for this transformation is proposed in **Scheme-II**. It is conceivable that a *ortho*-quinone methides intermediate **5** is initially formed in the presence of $HClO_4$-SiO_2, intermediates **6** are then formed in the second step, which then undergo dehydration to give the final products **4** (**Scheme-II**). In 2-naphthol the electron density at the benzylic C-1 position (which is in conjugation with the aromatic ring) is higher than that at the C-3 position. Thus the regioselective formation of the *ortho*-quinone methide from this compound involving the C-1 and C-2 positions is favoured. In simple phenolic compounds and 1-naphthol (which are weaker nucleophiles compared to 2-naphthol) the electron density at the ortho position of the hydroxyl group is not sufficient for the reaction of these compounds with the aldehydes leading to the formation of the corresponding *ortho*-quinone methides.



Scheme-II

Conclusion

In conclusion, we have developed a simple and highly efficient practical method for one-pot synthesis of 8,10-dimethyl-8,12-dihydro-12-aryl-9*H*-naphtho[1',2':5,6] pyrano[2,3-d]pyrimidine-9,11(10*H*)-diones using $HClO_4$-SiO_2 under solvent-free conditions. The notable features of this procedure are mild reaction conditions, simple experimental procdure and excellent yields (84-95 %), which make it a useful and attractive process for the synthesis of 8,10-dimethyl-8,12-dihydro-12-aryl-9*H*-naphtho[1',2':5,6]pyrano[2,3-d]pyrimidine-9,11(10*H*)-diones.

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